

Synthetic and Kinetics Aspects of the Electrooxidation of 4- Methylanisol

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4-methyl anisole is oxidized, in a first stage to produce a cation radical that undergoes deprotonation. Although a single oxidation peak is reported in literature corresponding to the loss of two electrons, we have been able to split it into a couple of peaks for one electron each. The cation radical is deprotonated to form a neutral radical which oxidizes to a cationic species. Two kind of reactions can occur, depending upon the reaction conditions: Whether a nucleophilic attack of the acetonitrile on the intermediate species, or polymerization of 4-methyl anisole.

The following table resumes the analyses of products:

| Oxidation Potential V vs Ag/AgNO ₃ | Charge Q (C) | Mass deposit (mg) | Mass soluble product (mg) | % deposit | % Soluble product |
|---|--------------|-------------------|---------------------------|-----------|-------------------|
| 1.40 | 114 | 5 | 29 | 15 | 85 |
| 2.10 | 180 | 7 | 29 | 20 | 80 |

Chronocoulometry allowed us to calculate the number of electrons involved in the reaction.

Spectroelectrochemical experiments showed the formation of the cation radical of 4-methyl anisol, followed by deprotonation.

It was found that the soluble product and the deposit have the same basic structure and the difference is the number of monomeric units.

SEM experiments showed that the morphology of the polymer obtained on the electrode surface changes with temperature. Variation of temperature has allowed the understanding of several kinetic aspects of those reactions taking place at the electrode-solution interface. A decrease in temperature causes a morphological variation of the deposit; it changes from a "cauliflower" morphology to a homogeneously distributed deposit on the electrode surface. In other words, the temperature affects the extension of the film

ACKNOWLEDGMENTS:

The authors gratefully acknowledge CDCHT-ULA, Project C-77896, for financial support.

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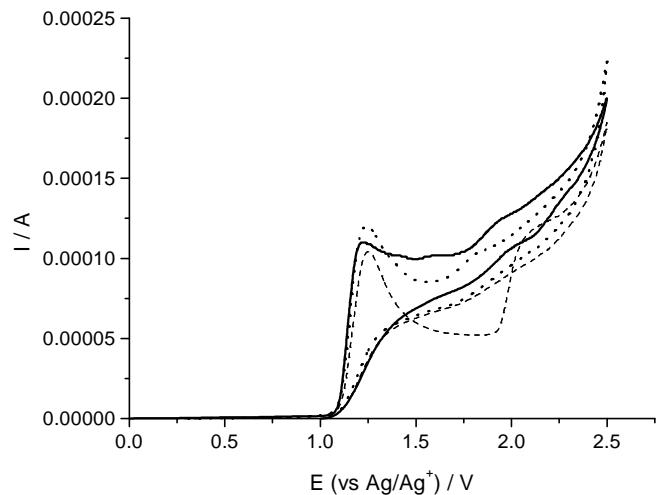


Fig. 1.- Cyclic Voltammetry of 4-methyl anisol in acetonitrile / Tetrabutyl ammonium Tetrafluoroborate at platinum electrode.

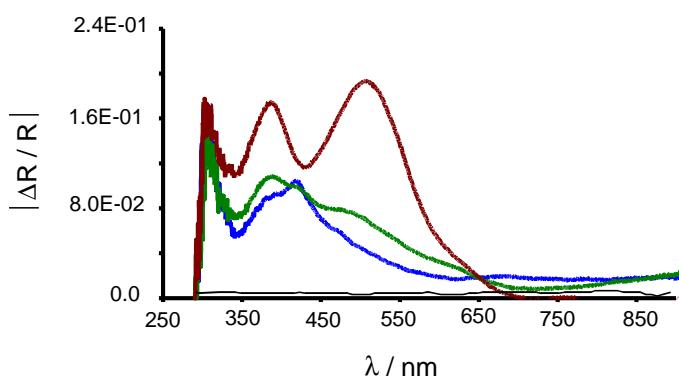


Fig. 2.- in situ UV-Visible spectra taken during the anodic oxidation of 4-methyl anisol

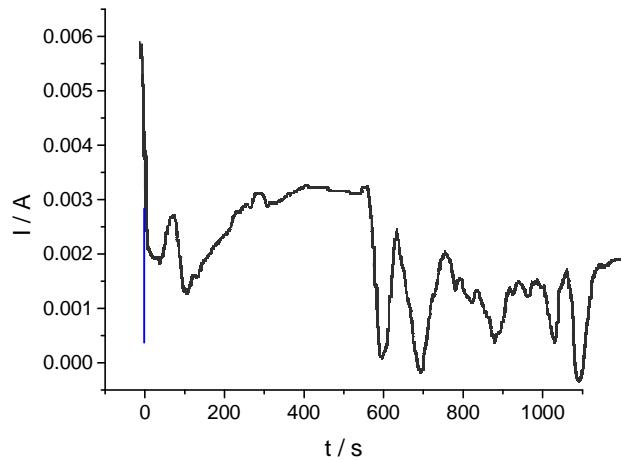


Fig. 3.- Chronoamperogram of 4-methyl anisol in CH₃CN